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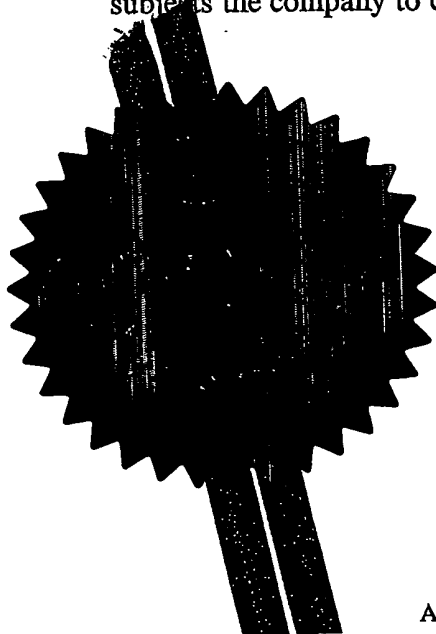
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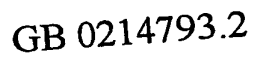
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Patents ADP number (if you know it)

2164246002

SECTION 30 (1977 ACT) APPLICATION FILED 23/8/02

If the applicant is a corporate body, give the country/state of its incorporation

United Kingdom

Title of the invention

BILIQUID FOAM ENTRAPMENT

Name of your agent (if you have one)

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BILIGUID FOAM ENTRAPMENT

The present invention relates to biliquid foam entrapment and, in particular, to a biliquid foam entrapped within a matrix of a polymeric material which is in the form of a discrete powder.

The entrapment of oils or oil soluble substances (especially perfumes and coloured dye precursors) in microcapsules and their subsequent coating onto paper and other surfaces is well known in the art. Microcapsules of this type comprise individual droplets of oil or oil soluble substances (of size ranging from sub-micrometre to tens of millimetres in diameter) around which polymer walls have been formed by one of a number of chemical processes. Usually such microcapsules are prepared as an aqueous suspension which is then capable, with the addition of suitable modifying reagents, of being sprayed or printed onto paper and other surfaces. The object in so doing is usually to prevent the evaporation of volatile substances (for example, perfumes) or the degradation or chemical reaction of oil soluble species (for example, colourless dye precursors) until the microcapsules are broken by the application of shear forces by scratching or scraping the coated surface with the consequent release of their contents. Such coatings find major uses, for example, in the forms of "scratch and sniff" perfume coatings or NCR (No Carbon Required) paper.

However, these microcapsules suffer from a number of disadvantages.

Firstly, the process by which microcapsules are formed is a lengthy and uncertain one in which control over temperature, pH and the absence of any form of contamination is essential. The formation of microcapsules, for example, by coacervation

gum acacia takes many hours and demands very close control of pH, temperature and cooling rate.

Similarly, the formation of microcapsule walls from aminoplast resins, such as melamine-formaldehyde or urea-formaldehyde takes at least eight hours during which precise control over all controllable parameters needs to be effected. Moreover, the effectiveness and completeness of any individual encapsulation process (and therefore the quality of the microcapsules so formed) depends largely on the chemical nature of the oil and/or oil soluble substances being encapsulated.

A further disadvantage of microcapsulation is that the thickness and therefore the strength of the microcapsule wall is variable and is not easily controllable and varies with the nature of the oil or oil-soluble substances being encapsulated. Thus microcapsules made by the same process but from different oils may have widely differing strengths and resistance to breakage during the printing process and during subsequent storage and use.

A yet further disadvantage of microencapsulation is the limited number of chemical processes and the limited number and type of polymeric wall materials which are available to form them. The choice as to the properties of the wall materials is consequently limited with regard to their flexibility, tensile strength, permeability, chemical inertness, mammalian toxicity and other properties including solubility and melting point (if any). In addition, some of the chemicals commonly used in the wall forming process are themselves highly irritating and may themselves be toxic such, for example, as the use or release of formaldehyde (a potential carcinogen) during the manufacture of aminoplast resin walls. Moreover, the remaining traces of formalin in the resulting microcapsule suspension are virtually impossible to eliminate to below acceptable levels for uses of

microcapsules and requires special precautions to be taken during the manufacturing process.

5 Whilst many of the processes to produce microcapsules produce dispersions of the microcapsules in a fluid medium, they can also be produced in the form of a powder.

10 Other methods of encapsulating oil within a powder are generally based upon the drying of an oil-in-water dispersion. Examples of this prior art include EP-B-0938932 which discloses a process for manufacturing a cosmetic and/or dermatological powder in which an oil-in-water dispersion comprising at least one modified starch is dehydrated to form a powder and US-A-6129906 in which a granular powder is formed by spray drying an aqueous dispersion of a silicone oil and a water-soluble carrier, the silicone oil being present in the dispersion as discrete droplets having a droplet size in the range of from 0.5 μ m to 20 μ m.

20 WO 99/05299 discloses a surface coating in which droplets of a non-polar substance are trapped within a polymer film, the surface coating being prepared by drying a dispersion of a film forming polymer containing droplets of a suspended biliquid foam or emulsion. Surface coatings only are disclosed and this reference does not teach the drying of the dispersions to form a powder.

25 We have now developed a discrete powder which is based upon the encapsulation of a biliquid foam.

30 Accordingly, in one aspect of the present invention provides a discrete powder which comprises particles in which a biliquid foam has been entrapped within a matrix of a polymeric material.

35 In another aspect the present invention provides a process for the preparation of a discrete powder which comprises a biliquid foam entrapped within a

comprises the steps of:

- i) preparing a biliquid foam,
- ii) forming a dispersion of the biliquid foam in an aqueous solution, suspension or dispersion of a polymeric material, and
- iii) subjecting the dispersion to drying under conditions such that a discrete powder is formed.

The discrete powder of the present invention is preferably produced by spray drying of the dispersion.

Biliquid foams are known in the art and are described in the following literature references by Sebba: "Biliquid foams", J. Colloid and Interface Science, 40 (1972) 468-474; and "The Behaviour of Minute Oil Droplets Encapsulated in a Water Film", Colloid Polymer Sciences, 257 (1979) 392-396. Neither of these articles suggest that biliquid foams might be used in the preparation of spray dried powders.

US Patent No. 4486333 to Sebba describes a particular method for the preparation of biliquid foams by agitating a hydrogen bonded liquid containing a soluble surfactant to produce a gas foam and intermittently adding to the gas foam a non-polar liquid which is immiscible with the hydrogen bonded liquid, the surfactant-containing hydrogen bonded liquid being selected to provide a spreading coefficient equal to or greater than zero.

The oil-based biliquid foam used in the spray dried powders of the present invention will preferably comprise from 70 to 95% by weight of the oil phase and from 5 to 30% by weight of the continuous phase. A surfactant to stabilise the biliquid foam may also be included in an amount of from 0.01 to 3%, preferably from 0.1 to 1% based on the total weight of the biliquid foam. The surfactant may dissolve in either the oil phase, the continuous phase or both phases of the biliquid foam.

Oils which may be used in the biliquid foam will in general be substantially water immiscible and liquid at room temperature and may be, for example, a cyclomethicone, dimethicone, phenyl trimethicone, dimethiconol, dimethicone copolyol, trimethylsiloxysilicate, an emollient ester such as isopropyl isostearate, lanolate, myristate or palmitate, or octyl palmitate, a glyceride such as avocado oil, coconut oil, soybean oil or sunflower oil, or a caprylic/capric triglyceride, a lanolin oil, orange oil, mineral oil or natural oil, or oleyl alcohol, or any other oil generally known for this purpose, or mixtures of the foregoing.

It will be understood that the oil phase of the biliquid foam may contain or consist of one or more active ingredients such as fragrances, flavours, perfumes, pharmaceuticals, sunscreens, dyes, sunstreams, pesticides etc.

The biliquid, foam may contain, as described above, a low level of a surfactant which may be, for example:-

a cationic surfactant such as an amidoamine, a quaternary ammonium compound or a sulphonium salt;

an amphoteric surfactant such as an acylamino-acid, an N-substituted alkylamine, an N-alkyl- β -aminopropionate, an N-alkylbetaine, an alkylimidazoline or a sulphobetaine;

an anionic surfactant such as an acyl-lactate, N-acylsarcosinate, alkyl-carboxylate (either mono- or polyvalent), alkyl ether carboxylate, N-alkyl-glutamate, fatty acid-peptide condensate, phosphated ethoxylated alcohol, alkyl sulphate, ethoxylated alkyl sulphate, alpha-olefin sulphonate or ester-linked sulphonate;

a nonionic surfactant such as an alkanolamide, amine oxides, ester of a polyhydric alcohol example as

or glycerol or a polyglycerol, or sorbitan, glucose or sucrose), a polyoxyethylene or polyoxypropylene derivative of an alcohol, amide or ester, or a polyoxyethylene/polyoxypropylene block copolymer;

5 or a suitable compatible mixture of these surfactants.

The continuous phase of the biliquid foam is generally an aqueous phase which may include therein a substantial level of a C_1-C_4 (water miscible) alcohol, or ethylene glycol or mixtures thereof.

10 The continuous phase of the biliquid foam may include therein preservatives, stabilizers or other materials known in the art.

Methods of producing biliquid foams are described in US-A-4486333 involving the preliminary formation of a gas foam in order to provide a sufficiently large surface area on which the biliquid foam can subsequently be formed. It has been found that the prior formation of a gas foam is not required to manufacture a stable biliquid foam, provided that a suitable stirring mechanism is provided in the manufacturing vessel. An aspect of the present invention is the ability to manufacture biliquid foams without the preliminary formation of gas foam, by the use of a tank incorporating a suitable stirring mechanism.

25 Such an apparatus comprises a tank provided with a stirrer in which the stirrer blade breaks the interface between the liquid and air. A delivery device is provided through which the oil phase (water immiscible liquid), which will comprise the internal phase of the dispersion is delivered to the tank. The design of the delivery device is such that the rate of addition of the internal phase fluid can be controlled and varied during the production process. A feature of the production process is that the internal (oil) phase is added to the stirred aqueous phase slowly at

first until sufficient droplets have been formed to constitute a large, additional surface area for the more rapid formation of new droplets. At this point, the rate of addition of the oil phase may be increased.

The production process consists of the following steps:

1. The addition of one or more chosen surfactants to one or other or both phases (as previously determined by experiment).
2. The charging of the aqueous phase into the bottom of a process vessel.
3. The incorporation of the stirrer into the vessel so that it stirs the surface of the aqueous phase.
4. Adjustment of the stirrer speed to a previously determined level.
5. The slow addition of the internal phase whilst continuing to stir at the prescribed speed.
6. The speeding up of the rate of addition of the oil phase once a prescribed amount (usually between 5% and 10% of the total amount to be added) has been added.

The stirring rate and the rate of addition of the oil phase are variables, the values of which depend upon the detailed design of the manufacturing plant (in particular, the ratio of tank diameter to impeller diameter), the physico-chemical properties of the oil phase and the nature and concentrations of the chosen surfactants. These can all be pre-determined by laboratory or pilot plant experiment.

It will be understood by those skilled in the art that other manufacturing methods may be used to produce the biliquid foams, as appropriate.

At the process termination the biliquid foam is

forms a discrete powder. Water-dispersible or water-soluble film forming polymers of many types are well known and include cellulose derivatives (for example, carboxymethylcellulose, hydroxyethylcellulose, cetylhydroxycellulose, hydroxypropylcellulose, hydroxypropylmethylcellulose, hydroxyethylmethylcellulose and methylcellulose), gelatin, gum arabic, gum acacia, gellan gum, shellac, carragenan, natural starches, modified starches, xanthan gums, alginates, dextrans, polyvinyl alcohol, polyvinyl pyrrolidone or polyamides and other water dispersible or water soluble film forming agents known in the art. The present invention may include the use of all the above singly or in combinations.

In carrying out the process of the present invention for forming a discrete powder the suspension of the biliquid foam in an aqueous solution, suspension or dispersion of the polymeric film is dried under conditions such that a discrete powder is formed. Preferably the said dispersion is spray dried. The choice of suitable spray drying conditions will be within the knowledge of a person skilled in the art and will depend upon various factors, including the melt temperature of the polymeric material, the amount of water contained in the dispersion, the ratio of polymeric material to the biliquid foam etc. Generally the inlet temperature for the spray dryer will be in the range of from 180° to 210°C and the outlet temperature will be in the range of from 85 to 110°C.

Typically, in carrying out the present invention the biliquid foam will have a droplet size in the range of from 5 to 45 micrometres. A biliquid foam having such a droplet size can generally be produced under low shear conditions. For the purpose of the present invention the mean droplet size of the biliquid foam should preferably be further reduced to

below 12 micrometres, for example by using higher shear conditions.

5 The biliquid foam is then mixed with an aqueous solution, suspension or dispersion of the polymeric material under conditions which generate a homogeneous dispersion. For example, using gentle stirring or using a high-shear device, such as a Roto Stator mixer.

10 It will be understood that although spray drying is the preferred method of producing the discrete powders of the present invention, other drying techniques, such as freeze drying can also be used.

15 The discrete powders of the present invention generally incorporate high levels of oil entrapped with the polymeric material, typically from 20 to 40% by weight based on the weight of the powder.

The discrete powders of the present invention will generally have a particle size in the range of from 5 to 100 μ m.

20 The invention provides a means of controlling the rate of release of the oil entrapped within the polymeric material by exercising control over the concentration and ratio to the biliquid foam of the film forming polymer in solution or suspension and
25 thereby controlling the thickness and strength of the film forming the outside of the particles.

The invention also allows for release of the oil by dissolution of the film by contact with water or other polar solvent. In addition, the water-soluble
30 or water dispersible film forming polymer may be partially or wholly crosslinked to render it partially or totally water insoluble by which means the rate of release of the entrapped biliquid foam may be controlled by the speed or absence of dissolution when
35 the powder makes contact with water or other polar

liquid in which it is otherwise insoluble.

It is to be understood that the invention is not limited to the specific details herein.

be such that it is sensitive to acidity or alkalinity so that the release of the entrapped oil may be determined by a change of pH or by the presence of another chemical species with which the film-forming polymers may react, so rendering it permeable or unstable. The powder may alternatively comprise a polymer which melts at a known and predetermined temperature to release the entrapped oil.

10 In one embodiment, the entrapped biliquid foam may comprise a perfume which, when dried into a discrete powder will behave and perform precisely as a conventional, microencapsulated "scratch and sniff" perfume as previously described. Furthermore, an encapsulating polymer may be chosen that allows the release of the perfume by diffusion over time, such as
15 in a room fragrancing device.

In another embodiment, a perfume or deodorising composition is entrapped according to this invention in a discrete powder which is incorporated into a
20 diaper or incontinence pad during manufacture so that the perfume or deodorising fluid is released on contact with water when the diaper or incontinence product is used, thereby masking or neutralising any disagreeable odour.

25 In another embodiment, the biliquid foam may be entrapped in water soluble polymer powder particles on a suitable applicator together with other reagents (for example, an abrasive material, such as a pumice or water soluble antimicrobial agents) to form a dry
30 surface which, when wetted, becomes an effective hard surface cleaning product.

In yet another embodiment, the matrix forming polymer may comprise a brittle film which ruptures easily when deformed so releasing the entrapped non-polar substance. In one application of this
35 embodiment, the powder may be coated onto a flexible film which may, for example, be shrunk onto the cap of

a consumer product such that if the flexible film is removed, the particles rupture so releasing the non-polar substance which, in this instance, may be the colourless precursor of a coloured dye which, on release, undergoes a chemical change to become highly coloured. This embodiment thereby gives a clear indication as to whether or not a closure has been tampered with.

The present invention will be further described with reference to the following Examples.

PREPARATION OF BILIQUID FOAMS

Preparation 1

A biliquid foam was prepared from the following ingredients.

| Ingredients | | Weight (g) | % |
|------------------------------------|--|------------|------|
| Aqueous Phase | | | |
| Water | | 396 | 9.9 |
| Sodium lauryl ether sulphate | | 4 | 0.1 |
| Oil Phase | | | |
| Volpo L3 | | 36.4 | 0.9 |
| Medium viscosity white mineral oil | | 3563.6 | 89.1 |
| Total | | 4000 | 100 |

The biliquid foam was prepared by adding the oil phase to the aqueous phase and stirring with a flat bladed stirrer at 300 rpm until the mean droplet size was 15-20 micrometres.

A 1kg sample was removed and this was stirred with a flat bladed stirrer at 500 rpm until the mean droplet size was 11 micrometres.

Preparation 2

| Ingredients | Weight (g) | % |
|----------------------|------------|-------|
| Aqueous Phase | | |
| Water | 148.5 | 9.9 |
| 5 Tween 20 | 1.5 | 0.1 |
| Oil Phase | | |
| PEG25 castor oil | 13.5 | 0.9 |
| KMC | 1269.7 | 84.65 |
| Pergascript Red I-6B | 66.8 | 4.45 |
| 10 Total | 1500.0 | 100 |

15 The biliquid foam was prepared by adding the oil phase to the aqueous phase and stirring with a flat bladed stirrer at 116 rpm. The mean droplet diameter was 35 micrometres. The stirrer speed was then increased to 250 rpm and stirred until the mean droplet size was less than 12 micrometres.

Preparation 3

| Ingredients | Weight (g) | % |
|------------------------------|------------|------|
| Aqueous Phase | | |
| Water | 47.67 | 9 |
| Sodium lauryl ether sulphate | 0.53 | 0.1 |
| Oil Phase | | |
| Laureth 3 | 4.77 | 0.9 |
| Dow Corning 200 50cst | 476.74 | 90.0 |
| 25 Total | 529.71 | 100 |

30 The biliquid foam was prepared by adding the oil phase to the aqueous phase whilst stirring with a flat bladed stirrer at 200 rpm for 45 minutes.

Preparation 4

| Ingredients | Weight (g) | % |
|-----------------------------------|------------|-------|
| Aqueous Phase | | |
| Water | 44.97 | 9 |
| 5 Sodium lauryl ether sulphate | 0.5 | 0.1 |
| Kathon 1CG II | 0.03 | 0.006 |
| Oil Phase | | |
| Oleth 10 | 4.5 | 0.9 |
| 10 Orange oil | 450.0 | 90.0 |
| Total | 500 | 100 |

15 The biliquid foam was prepared by adding the oil phase to the aqueous phase whilst stirring with a flat bladed stirrer at 200 rpm for 45 minutes.

Preparation 5

| Ingredients | Weight (g) | % |
|------------------------------------|------------|--------|
| Aqueous Phase | | |
| 20 Water | 52.60 | 9.8 |
| Sodium lauryl ether sulphate | 0.532 | 0.1 |
| Kathon 1CG II | 0.026 | 0.0048 |
| Oil Phase | | |
| 25 Etocas 25 (PEG25 Castor oil) | 4.78 | 0.9 |
| Rose oil fragrance | 478.44 | 89.2 |
| L301844 | ----- | |
| Total | 536.378 | 100 |

30 The biliquid foam was prepared by adding the oil phase to the aqueous phase whilst stirring with a flat bladed stirrer at 200 rpm for 45 minutes.

Preparation 6

| Ingredients | Weight (g) | % |
|-------------------------|------------|------|
| Aqueous Phase | | |
| Water | 14.85 | 9.9 |
| Tween 20 | 0.15 | 0.1 |
| Oil Phase | | |
| Oleth 10 | 1.35 | 0.9 |
| Octyl methoxy cinnamate | 133.65 | 89.1 |
| Total | 150 | 100 |

The biliquid foam was prepared by adding the oil phase to the aqueous phase whilst stirring with a flat bladed stirrer at 200 rpm for 45 minutes.

Preparation 7

| Ingredients | Weight (g) | % |
|-------------------------|------------|------|
| Aqueous Phase | | |
| Water | 11.29 | 8.79 |
| Tween 20 | 0.26 | 0.20 |
| Oil Phase | | |
| PEG25 castor oil | 0.64 | 0.5 |
| Oleth 10 | 0.64 | 0.5 |
| Household Fragrance oil | 115.55 | 90 |
| Total | 128.38 | 100 |

The biliquid foam was prepared by adding the oil phase to the aqueous phase whilst stirring with a flat bladed stirrer at 200 rpm for 45 minutes.

Preparation 8

| | Ingredients | Weight (g) | % |
|----|-----------------------|------------|-------|
| | Aqueous Phase | | |
| | Water | 9 | 9 |
| 5 | Laureth 23 | 1 | 1 |
| | Oil Phase | | |
| | Gransil GCM-5 | 49.24 | 49.24 |
| | Cetearyl isonanoate | 7.78 | 7.78 |
| | Isopar K | 7.78 | 7.78 |
| 10 | Dow Corning 200 50cst | 0.97 | 0.97 |
| | Gransil DMCM-5 | 24.25 | 24.25 |
| | Total | 100 | 100 |

15 The biliquid foam was prepared by adding the oil phase to the aqueous phase and stirring with a flat bladed stirrer at 174 rpm. The stirrer speed was increased to 300 rpm to help with the inclusion of the oil before continuing to stir at 174 rpm until the mean droplet size was 11 rpm.

20

Preparation 9

| | Ingredients | Weight (g) | % |
|----|----------------------|------------|------|
| | Aqueous Phase | | |
| | Water | 9.9 | 9.9 |
| 25 | Tween 20 | 0.1 | 0.1 |
| | Oil Phase | | |
| | Ibuprofen | 4.5 | 4.5 |
| | Isopropyl myristate | 84.5 | 84.5 |
| | Laureth 3 | 1 | 1 |
| 30 | Total | 100 | 100 |

35 The biliquid foam was prepared by adding the oil phase (ibuprofen fully dissolved in the isopropyl myristate) to the aqueous phase and stirring with a flat bladed stirrer at 174 rpm. The preparation was stirred after the inclusion of the oil until the mean droplet size was 11 rpm.

Preparation of Dispersions and Spray Drying

EXAMPLE 1

5 The dispersion was prepared by stirring the
bilibiquid foam into the aqueous polymer immediately
before spray-drying.

| Ingredients | Weight (g) | % |
|--|------------|------|
| Preparation 1 | 76.9 | 7.7 |
| Gum acacia (30% by weight in demineralized water) | 923.1 | 92.3 |
| Total | 1000 | 100 |

| Spray drying conditions | |
|--------------------------------------|---|
| Pilot plant | Tests were carried out in a 1m diameter pilot spray drying tower with downward co current air flow. Atomisation was carried out with a two fluid nozzle. |
| Total non volatiles | 34.6% |
| Oil: polymer (dry basis) | 20:80 |
| Inlet/outlet temperature | 200°C/95°C |
| Yield | 85.2% |
| Comment | |
| Product Characterisation | |
| Nature of dry particle | Fine powder |
| Oil encapsulation | Good |
| Oil release | Moderate amount of loose oil visible on release. |
| Mean droplet size before spraying | 1.99µm |

EXAMPLE 2

The dispersion was prepared by stirring the biliquid foam into the aqueous polymer immediately before spray drying.

| Ingredients | Weight (g) | % |
|---|--------------|------------|
| Preparation 1 | 73.85 | 8.7 |
| PVP K30 (30% by weight in demineralized water) | 465.9 | 54.8 |
| Mowiol (5% by weight in demineralized water) | 310.6 | 36.5 |
| Total | 850.4 | 100 |

| Spray drying conditions | |
|-----------------------------------|--|
| Pilot plant | Tests were carried out in a 1m diameter pilot spray drying tower with downward co current air flow. Atomisation was carried out with a two fluid nozzle. |
| Total non volatiles | 26% |
| Oil: polymer (dry basis) | 30:70 |
| Inlet/outlet temperature | 210°C/110°C |
| Yield | about 100% |
| Comment | |
| Product Characterisation | |
| Nature of dry-particle | Good |
| Oil encapsulation | Good |
| Oil release | Little visible oil. |
| Mean droplet size before spraying | 6.1µm peak at 11µm. |

EXAMPLE 3

The dispersion was prepared by stirring the biliquid foam and water into the aqueous polymer immediately before spray drying.

| 5 | Ingredients | Weight (g) | % |
|----|---|-------------------|----------|
| | Preparation 1 | 100 | 11.7 |
| | Water | 74.64 | 8.7 |
| | Maltodextrin (40% by weight in demineralized water) | 52.5 | 6.1 |
| 10 | PVP k30 (30% by weight in demineralized water) | 630 | 73.5 |
| | Total | 847.14 | 100 |

| | | |
|----|-----------------------------------|--|
| 15 | Spray drying conditions | |
| | Pilot plant | Tests were carried out in a 1m diameter pilot spray drying tower with downward co current air flow. Atomisation was carried out with a two fluid nozzle. |
| | Total non volatiles | 35% |
| | Oil: polymer (dry basis) | 30:70 |
| | Inlet/outlet temperature | 185°C/85°C increased to 90°C |
| | Yield | 17.2% |
| 20 | Comment | Product slightly damp initially but spray dried well with higher outlet temperature. |
| | Product Characterisation | |
| | Nature of dry particle | Good |
| | Oil encapsulation | Good |
| | Oil release | Slight amount of loose oil visible. |
| 25 | Mean droplet size before spraying | 1.2µm peak at 9µm. |

EXAMPLE 4

The dispersion was prepared by stirring the biliquid foam and make up water into the aqueous polymer immediately before spray drying.

| Ingredients | Weight (g) | % |
|--|------------|-------|
| Preparation 2 | 116.67 | 11.67 |
| Water | 66.67 | 6.67 |
| PVP K30 (30% by weight in demineralized water) | 816.67 | 81.67 |
| Total | 1000 | 100 |

| Spray drying conditions | |
|-----------------------------------|--|
| Pilot plant | Tests were carried out in a 1m diameter pilot spray drying tower with downward co current air flow. Atomisation was carried out with a two fluid nozzle. |
| Total non volatiles | 35% |
| Oil: polymer (dry basis) | 30:70 |
| Inlet/outlet temperature | 203°C/95°C |
| Yield | 64.21% |
| Comment | |
| Product Characterisation | |
| Nature of dry particle | Good |
| Oil encapsulation | Good |
| Oil release | Little visible free oil. |
| Mean droplet size before spraying | 0.58 μ m, peaks at 0.15, 0.7 and 12 μ m. |

EXAMPLE 5

The dispersion was prepared by stirring the biliquid foam and water into the aqueous polymers immediately before spray drying.

| Ingredients | Weight (g) | % |
|---|--------------|------------|
| Preparation 3 | 89.9 | 11.7 |
| Water | 67.1 | 8.7 |
| Maltodextrin (40% by weight in demineralized water) | 47.2 | 6.1 |
| PVP k30 (30% by weight in demineralized water) | 566.6 | 73.5 |
| Total | 770.9 | 100 |

| Spray drying conditions | |
|-----------------------------------|--|
| Pilot plant | Tests were carried out in a 1m diameter pilot spray drying tower with downward co current air flow. Atomisation was carried out with a two fluid nozzle. |
| Total non volatiles | 35% |
| Oil: polymer (dry basis) | 30:70 |
| Inlet/outlet temperature | 195°C/95°C |
| Yield | 56.6% |
| Comment | Spray dried well |
| Product Characterisation | |
| Nature of dry particle | Good |
| Oil encapsulation | Good |
| Oil release | No visible oil on surface |
| Mean droplet size before spraying | 9.9µm |

EXAMPLE 6

The dispersion was prepared by stirring the
billyquid foam and water into the aqueous polymers
immediately before spray drying.

| Ingredients | Weight (g) | % |
|---|--------------|------------|
| Preparation 4 | 105.4 | 13.7 |
| Water | 48.8 | 6.3 |
| Maltodextrin (40% by weight in demineralized water) | 614.7 | 79.9 |
| Total | 768.9 | 100 |

| Spray drying conditions | |
|-----------------------------------|--|
| Pilot plant | Tests were carried out in a 1m diameter pilot spray drying tower with downward co current air flow. Atomisation was carried out with a two fluid nozzle. |
| Total non volatiles | 45% |
| Oil: polymer (dry basis) | 27.8 : 72.2 |
| Inlet/outlet temperature | 195°C/95°C |
| Yield | about 100% |
| Comment | Spray dried well |
| Product Characterisation | |
| Nature of dry particle | Good |
| Oil encapsulation | Good |
| Oil release | Little visible oil. |
| Mean droplet size before spraying | 1.4µm |

EXAMPLE 7

The dispersion was prepared by stirring the
bilibiquid foam and water into the aqueous polymers
immediately before spray drying.

| Ingredients | Weight (g) | % |
|---|--------------|------------|
| Preparation 4 | 101.7 | 11.7 |
| Water | 147.1 | 16.9 |
| Maltodextrin (40% by weight in demineralized water) | 266.9 | 30.6 |
| Gum acacia | 355.9 | 40.8 |
| Total | 871.6 | 100 |

| Spray drying conditions | |
|-----------------------------------|--|
| Pilot plant | Tests were carried out in a 1m diameter pilot spray drying tower with downward co current air flow. Atomisation was carried out with a two fluid nozzle. |
| Total non volatiles | 35% |
| Oil: polymer (dry basis) | 30:70 |
| Inlet/outlet temperature | 195°C/95°C |
| Yield | 78.3% |
| Comment | Spray dried well |
| Product Characterisation | |
| Nature of dry particle | Good |
| Oil encapsulation | Good |
| Oil release | Little visible oil at surface. |
| Mean droplet size before spraying | 1.3µm |

EXAMPLE 8

The dispersion was prepared by stirring the biliquid foam and water into the aqueous polymers immediately before spray drying.

| Ingredients | Weight (g) | % |
|---|--------------|------------|
| Preparation 5 | 81.4 | 11.7 |
| Water | 89.3 | 12.8 |
| Maltodextrin (40% by weight in demineralized water) | 128.3 | 18.4 |
| PVP k30 (30% by weight in demineralized water) | 399.1 | 57.2 |
| Total | 698.1 | 100 |

| Spray drying conditions | |
|-----------------------------------|--|
| Pilot plant | Tests were carried out in a 1m diameter pilot spray drying tower with downward co current air flow. Atomisation was carried out with a two fluid nozzle. |
| Total non volatiles | 35% |
| Oil: polymer (dry basis) | 30:70 |
| Inlet/outlet temperature | 195°C/95°C |
| Yield | 66.1% |
| Comment | Spray dried well |
| Product Characterisation | |
| Nature of dry particle | Good |
| Oil encapsulation | Good |
| Oil release | No visible oil at surface |
| Mean droplet size before spraying | 0.95µm peaks at 1µm and 6.5µm |

EXAMPLE 9

The dispersion was prepared by stirring the biliquid foam and water into the aqueous polymer immediately before spray drying.

| Ingredients | Weight (g) | % |
|---|---------------|------------|
| Preparation 6 | 100 | 11.7 |
| Water | 74.64 | 8.7 |
| Maltodextrin (40% by weight in demineralized water) | 52.5 | 6.1 |
| PVP k30 (30% by weight in demineralized water) | 630 | 73.5 |
| Total | 857.14 | 100 |

| Spray drying conditions | |
|-----------------------------------|--|
| Pilot plant | Tests were carried out in a 1m diameter pilot spray drying tower with downward co current air flow. Atomisation was carried out with a two fluid nozzle. |
| Total non volatiles | 35% |
| Oil: polymer (dry basis) | 30:70 |
| Inlet/outlet temperature | 175°C/95°C |
| Yield | 92% |
| Comment | Spray dried well |
| Product Characterisation | |
| Nature of dry particle | Good |
| Oil encapsulation | Good |
| Oil release | Minimal free oil visible on surface. |
| Mean droplet size before spraying | 0.7µm, peak at 10µm |

EXAMPLE 10

The dispersion was prepared by stirring the biliquid foam and water into the aqueous polymers immediately before spray drying.

| Ingredients | Weight (g) | % |
|---|--------------|------------|
| Preparation 7 | 89.9 | 11.7 |
| Water | 67.1 | 8.7 |
| Maltodextrin (40% by weight in demineralized water) | 47.2 | 6.1 |
| PVP k30 (30% by weight in demineralized water) | 566.6 | 73.5 |
| Total | 770.9 | 100 |

| | |
|-----------------------------------|--|
| Spray drying conditions | |
| Pilot plant | Tests were carried out in a 1m diameter pilot spray drying tower with downward co current air flow. Atomisation was carried out with a two fluid nozzle. |
| Total non volatiles | 35% |
| Oil: polymer (dry basis) | 30:70 |
| Inlet/outlet temperature | 195°C/90°C |
| Yield | 93.8% |
| Comment | Spray dried well |
| Product Characterisation | |
| Nature of dry particle | Good |
| Oil encapsulation | Good |
| Oil release | Little visible oil on surface. |
| Mean droplet size before spraying | 2.39 μ m peaks at 1.5 μ m and 7.5 μ m |

EXAMPLE 11

The dispersion was prepared by stirring the biliquid foam and water into the aqueous polymers immediately before spray drying.

| Ingredients | Weight (g) | % |
|---|------------|------------|
| Preparation 8 | 67.2 | 11.3 |
| Water | 50.2 | 8.5 |
| Maltodextrin (40% by weight in demineralized water) | 53.3 | 9.0 |
| PVP k30 (30% by weight in demineralized water) | 423.3 | 71.3 |
| Total | 594 | 100 |

| Spray drying conditions | |
|-----------------------------------|--|
| Pilot plant | Tests were carried out in a 1m diameter pilot spray drying tower with downward co current air flow. Atomisation was carried out with a two fluid nozzle. |
| Total non volatiles | 35% |
| Oil: polymer (dry basis) | 29:71 |
| Inlet/outlet temperature | 195°C/95°C |
| Yield | 82.3% |
| Comment | Spray dried well |
| Product Characterisation | |
| Nature of dry particle | Good |
| Oil encapsulation | Good |
| Oil release | No visible free oil |
| Mean droplet size before spraying | 7.26µm, peak at 11µm |

EXAMPLE 12

The dispersion was prepared by stirring the biliquid foam and water into the aqueous polymers immediately before spray drying.

| | | | |
|----|---|-------------------|------------|
| 5 | Ingredients | Weight (g) | % |
| | Preparation 9 | 79.1 | 11.7 |
| | Water | 59.1 | 8.7 |
| | Maltodextrin (40% by weight in demineralized water) | 41.5 | 6.1 |
| 10 | PVP k30 (30% by weight in demineralized water) | 498.5 | 73.5 |
| | Total | 678.1 | 100 |

| | | |
|----|-----------------------------------|--|
| 15 | Spray drying conditions | |
| | Pilot plant | Tests were carried out in a 1m diameter pilot spray drying tower with downward co current air flow. Atomisation was carried out with a two fluid nozzle. |
| | Total non volatiles | 35% |
| | Oil: polymer (dry basis) | 30:70 |
| | Inlet/outlet temperature | 195°C/98°C |
| | Yield | 76.5% |
| 20 | Comment | Spray dried well |
| | Product Characterisation | |
| | Nature of dry particle | Good |
| | Oil encapsulation | Good |
| | Oil release | Minimal free oil visible on surface. |
| 25 | Mean droplet size before spraying | 18.71µm |

Footnote to the Examples

| | Trade Name | Supplier | INCI Name |
|----|--------------------------|---------------------------------------|---|
| 5 | Dow Corning 200 50cst | Dow Corning | Silicone |
| | Etocas 25 | Croda Chemicals | PEG-25 Castor Oil |
| | Gransil DMCM-5 | Grant Chemicals | Cyclopentasiloxane (D5) (and) Polysilicone- 11 (and) Dimethicone. (An Organopolysiloxane mixture) |
| | Gransil GCM-5 | Grant Chemicals | Cyclopentasiloxane (D5) (and) Polysilicone- 11 (An Organopoly- siloxane mixture) |
| | Isopar K | Exxon Chemical Ltd | Isoparaffin |
| 10 | Kathon ICG 11 | Chesham Chemicals Limited | Mixture of: 5-chloro 2-methyl-4-isothia- zolin-3-one and 2- methyl-4-isothiazolin- 3-one |
| | KMC | Rutgers Kureha Solvents GmbH | Diisopropylnaphthalene isomers (mixture) |
| | Mowiol 4-88 | Kuraray Specialties Europe | Polyvinyl alcohol, partly saponified |
| | Pergascript red I-6B | Ciba Specialties | Bisindolyolphthalide compound |
| 15 | Tween 20 | Fisher Chemicals | Polysorbate 20 |

WE CLAIM:

1. A discrete powder which comprises particles
in which a biliquid foam has been entrapped within a
5 matrix of a polymeric material.

2. A powder as claimed in claim 1 which is a
spray dried powder.

10 3. A powder as claimed in claim 1 or claim 2
which has a particle size in the range of from 5 to
100 μ m.

15 4. A powder as claimed in any one of the
preceding claims wherein the polymeric material
encapsulating the biliquid foam is selected from
carboxymethylcellulose, hydroxyethylcellulose, cetyl-
hydroxycellulose, hydroxypropylcellulose, hydroxy-
propylmethylcellulose, hydroxyethylmethylcellulose
20 methylcellulose, gelatin, gum arabic, gum acacia,
gellan gum, shellac, carragenan, natural starch,
modified starch, xanthan gum, an alginate, a dextrin,
polyvinyl alcohol, polyvinylpyrrolidone or a
polyamide, or mixtures thereof.

25 5. A powder as claimed in any one of the
preceding claims wherein the biliquid foam comprises
an substantially water immiscible internal oil phase
which comprises a cyclomethicone, dimethicone, phenyl
30 trimethicone, di-methiconol, dimethicone copolyol,
trimethylsiloxysilicate, isopropyl isostearate,
lanolate, myristate or palmitate, or octyl palmitate,
avocado oil, coconut oil, soybean oil or sunflower
oil, a caprylic/capric triglyceride, a lanolin oil,
35 orange oil, mineral oil or natural oil, or oleyl
alcohol or mixtures thereof.

6. A powder as claimed in claim 5 which comprises from 20% to 40% by weight of an oil, based upon the weight of the powder.

5 7. A process for the preparation of a discrete powder which comprises a biliquid foam entrapped within a matrix of a polymeric material, which process comprises the steps of:

- 10 i) preparing a biliquid foam,
ii) forming a dispersion of the biliquid foam in an aqueous solution, suspension or dispersion of a polymeric material, and
15 iii) subjecting the dispersion to drying under conditions such that a discrete powder is formed.

8. A process as claimed in claim 7 wherein the drying is carried out by spray drying of the dispersion.

20 9. A process as claimed in claim 7 or claim 8 wherein the biliquid foam prepared in step (i) has a droplet size in the range of from 5 to 40 micrometres.

25 10. A process as claimed in claim 7 wherein the biliquid foam has a droplet size of below 12 micrometres.

30 11. A process as claimed in any one of claims 7 to 9 wherein the polymeric material is selected from selected from carboxymethylcellulose, hydroxyethyl-cellulose, cetylhydroxycellulose, hydroxypropyl-cellulose, hydroxypropylmethylcellulose, hydroxyethyl-methylcellulose, methylcellulose, gelatin, gum arabic,
35 gum acacia, gellan gum, shellac, carragenan, natural starch, modified starch, xanthan gum, an alginate, a dextrin, polyvinyl alcohol, polyvinylpyrrolidone or a

polyamide, or mixtures thereof.

12. A process as claimed in any one of claims 7
to 11 wherein the biliquid foam comprises an
5 essentially water immiscible internal oil phase which
comprises a cyclomethicone, di-methicone, phenyl
trimethicone, dimethiconol, di-methicone copolyol,
trimethylsiloxysilicate, isopropyl isostearate,
lanolate, myristate or palmitate, octyl palmitate,
10 avocado oil, coconut oil, soybean oil or sunflower
oil, a caprylic/capric tri-glyceride, a lanolin oil,
orange oil, mineral oil or natural oil, or oleyl
alcohol, or mixtures thereof.

13. A process as claimed in any one of claims 7
to 11 wherein the continuous phase of the biliquid
foam is an aqueous phase.

14. A process as claimed in any one of claims 7
to 13 wherein the aqueous phase includes therein a C₁-
20 C₄ alcohol or ethylene glycol.

15. A process as claimed in any one of claims 7
to 13 wherein the spray drying conditions comprise an
25 inlet temperature in the range of from 180 to 210°C
and an outlet temperature in the range of from 85 to
100°C.

16. A process as claimed in any one of claims 7
to 15 wherein the discrete powder has a particle size
30 in the range of from 5 to 100µm.

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